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STATUS OF
REFRACTORY METALS SHEET ROLLING PANEL



NO OTS

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The National Academy of Sciences—National Research Council is a private, nonprofit organization of scientists, dedicated to the furtherance of science and to its use for the general welfare.

The Academy itself was established in 1863 under a Congressional charter signed by President Lincoln. Empowered to provide for all activities appropriate to academies of science, it was also required by its charter to act as an adviser to the Federal Government in scientific matters. This provision accounts for the close ties that have always existed between the Academy and the Government, although the Academy is not a governmental agency.

The National Research Council was established by the Academy in 1916, at the request of President Wilson, to enable scientists generally to associate their efforts with those of the limited membership of the Academy in service to the nation, to society, and to science at home and abroad. Members of the National Research Council receive their appointments from the President of the Academy. They include representatives nominated by the major scientific and technical societies, representatives of the Federal Government, and a number of members-at-large. In addition, several thousand scientists and engineers take part in the activities of the Research Council through membership on its various boards and committees.

Receiving funds from both public and private sources, by contributions, grant, or contract, the Academy and its Research Council thus work to stimulate research and its applications, to survey the broad possibilities of science, to promote effective utilization of the scientific and technical resources of the country, to serve the Government, and to further the general interests of science.

MATERIALS ADVISORY BOARD

The Materials Advisory Board is a part of the Division of Engineering and Industrial Research of the Academy-Research Council. It was organized in 1951 under the name of the Metallurgical Advisory Board, with assignments from the then existing Research and Development Board of the Department of Defense. At that time, the Research and Development Board requested the Board to accept tasks covering a broad spectrum of metallurgical science and technology as related to the Armed Services, and to include certain other areas such as collection and dissemination of information, and cooperation with professional societies in publication of significant metallurgical data.

Since the organization date, the above scope has been expanded to include organic and inorganic nonmetallic materials, and the name has been changed to the Materials Advisory Board. Concurrently, the Board's membership, staff, and operations have been adjusted to encompass the greater diversity of materials and to concentrate on materials research and development, excluding other activities except to the extent that they support and strengthen the Board's fulfillment of its primary responsibility.

The Office of the Director of Defense Research and Engineering, Office of the Secretary of Defense, is the government agency which now requests specific consulting and advisory services under this broadened program. Under a contract between the Office of the Secretary of Defense and the National Academy of Sciences, the Board's assignment is:

"... at the written request of the Director of Defense Research and Engineering, or his designated representative, to conduct studies, surveys, make critical analyses, and prepare and furnish to the Director of Defense Research and Engineering advisory and technical reports, with respect to the entire field of materials research, including the planning phases thereof; and shall, in addition, perform such other services as may be agreed upon in writing, from time to time, by the Director of Defense Research and Engineering and the President of the Contractor.

"Task assignments under this contract will be as mutually agreed by the Director of Defense Research and Engineering or his designated representative and the Contractor. Recommendations for tasks may be proposed to the Director of Defense Research and Engineering by agencies of the Military Departments, the Office of the Secretary of Defense, or the Contractor."

NATIONAL ACADEMY OF SCIENCES
NATIONAL RESEARCH COUNCIL

MATERIALS ADVISORY BOARD
OF THE
DIVISION OF ENGINEERING AND INDUSTRIAL RESEARCH

December 1, 1962

MAILING ADDRESS:
2101 CONSTITUTION AVENUE, N.W.
WASHINGTON 25, D. C.

OFFICES:
1155 16TH STREET, N.W.

Dear Sir:

I am forwarding herewith a report entitled "Status of Refractory Metals Sheet Rolling Panel," which has been submitted through the National Academy of Sciences-National Research Council to the Director of Defense Research and Engineering. This report has been reviewed by the Refractory Metals Sheet Rolling Panel and by individual members of the Materials Advisory Board who have competence in the field.

In accordance with an agreement with the Office of the Director of Defense Research and Engineering, this report is being distributed on the same date it is being transmitted to the Department of Defense. Therefore, as of this date, it has not been reviewed by the Office of the Director of Defense Research and Engineering.

Very truly yours,



C. S. Marvel, Chairman
Materials Advisory Board

Enclosure

STATUS OF
REFRACTORY METALS SHEET ROLLING PANEL

Joseph R. Lane
and
G. Mervin Ault

Materials Advisory Board
Division of Engineering and Industrial Research

National Academy of Sciences
National Research Council
Washington 25, D.C.

December 1, 1962

No Portion of this Report may be Published
without Prior Approval of the Contracting
Agency.

This Report Prepared and Submitted to the
Office of the Director of Defense Research
and Engineering under ARPA Contract SD-118,
Between the Department of Defense and the
National Academy of Sciences.

REFRACTORY METALS SHEET ROLLING PANEL ACTIVITIES

October 15, 1962

It is the purpose of this report to describe the functions and current status (as of August 1962) of the activities of the Materials Advisory Board Refractory Metals Sheet Rolling Panel. For this discussion, the term "refractory" metals includes molybdenum, columbium, tantalum, and tungsten. An earlier report of this kind (dated May 1961) has been issued. Because of the importance of refractory metals in the defense effort, and the impact of this program on the metals industry, it seemed desirable to bring the earlier account up to date. The objectives remain unchanged, but there is now considerable progress to report.

With operating temperatures above 1900 F, and with stresses above 15,000 psi, it is probable that refractory metals will be required and it is almost a certainty at temperatures of 2200 F and above, regardless of stress levels. An alternative approach is cooling, but usually a severe penalty is paid, and often an impossible penalty in weight and complexity. Those applications for which graphite, ceramics, or ablating plastics have proven to be suitable are quite limited. The initial requirements for refractory materials a few years ago were for advanced propulsion devices for aircraft - the ramjet and the turbojet. Now they are required in the nozzles of the solid and liquid propelled rocket and, with the advent of the space age, they are essential in some re-entry vehicles, space power systems, and perhaps in the nuclear rocket. Many of the requirements involve sheet. Certainly sheet has been required for structures in the ramjet,

and sheet is now required for the surfaces of re-entry glide vehicles and components of space power systems.

The program was created because of the need to accelerate efforts to achieve high-quality, consistent sheet products from refractory metals and their alloys. Only six years ago, essentially no research had been conducted on alloys of columbium, tantalum, or tungsten for use as structural materials at high temperatures. Only molybdenum had been studied for this purpose to any degree. Some of the important shortcomings of alloys available when the program started were:

1. Inconsistent properties within a sheet and from sheet to sheet.
2. Poor surface quality and flatness.
3. Tendency to delaminate.
4. High and variable ductile-brittle transition temperature.
5. Lack of availability in large sizes and thin gages.

It was clear that government sponsorship of this development was necessary for several reasons. First, the potential market for refractory metal sheet products would probably not be large enough to make such an expensive private development profitable. The days when each vehicle was purchased in large quantities, thus guaranteeing a stable market for a long period of time, are evidently gone, probably forever. Most military or space vehicles will be purchased in quantities of less than one hundred; more probably, a tenth of that figure. Also, it cannot be stated in advance that only one, or even only two, of the refractory metals will be required. Each may find its place, and thus we may find that at least one, and in some cases,

several, of each system may be needed. Thus, several alloys may be required, but no one in sufficient amounts to justify extensive private sponsorship. Most important, technology must be developed at an accelerated rate to meet defense requirements.

Government sponsorship has the concomitant requirement that all of the information developed be published, to insure that the information developed is available to all who may have a legitimate requirement for it. This is tremendously important. All processing data that can be written down to permit other organizations to duplicate the product will be released, so that others can have a clearly defined base from which to initiate their own developments.

The Department of Defense initiated a Refractory Metals Sheet Program in June of 1959. The Navy Bureau of Naval Weapons manages the contract phases of many of the programs. The Manufacturing Technology Laboratories of the Air Force is also participating. The Materials Advisory Board was requested by DOD to form an Advisory Panel to assist in the technical aspects of the program. The organization is shown diagrammatically in Figure 1.

From the beginning, it has been fairly well accepted that the refractory sheet program for each material will be divided into phases as follows:

- PHASE I (a) Development of optimum production techniques, and production of sheet under controlled conditions to establish uniformity and quality.

(b) Production of quantity required for Phase II,
III, and IV.

Phase II Establishment of Preliminary Design Data.

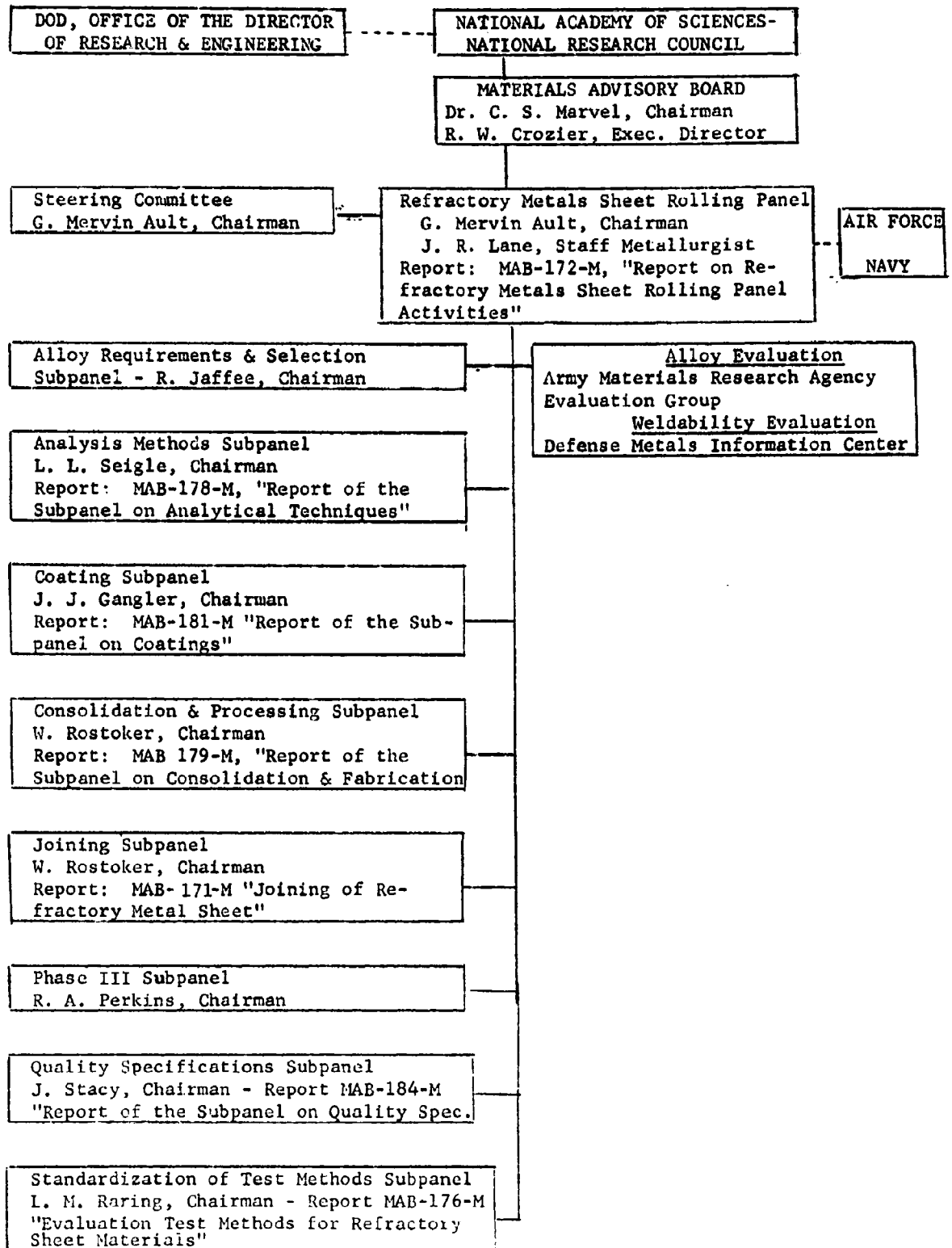
Phase III Establishment of standards and procedures for evaluation of the sheet, establishment of forming and joining limits and procedures, tests of fabricated structural elements, and finally, if necessary, the design and evaluation of prototype aerospace vehicle or power plant components.

Phase IV Determination of all Required Design Data.

The panel has found it necessary to form several subpanels or ad hoc working groups to carry out its responsibilities, and thus an understanding of the activity can best be obtained by referring to the following list of subpanels:

- Alloy Requirements and Selection
- Chemical Analysis
- Coatings
- Consolidation and Processing
- Joining
- Phase III (Fabricability Evaluation)
- Quality Specifications
- Testing Standards

The first problem has been to decide which alloys should be in the program by determining the requirements for the refractory metal sheet, and then by selecting the metals and alloys that might meet these requirements. This has been the responsibility of the Subpanel on Alloy Requirements and Selection. They have surveyed the need for refractory metals by consulting



many of the consumers. They have also conducted a preliminary state-of-the-art survey to learn what alloys of refractory metals have been developed in this country. Based upon these surveys, they decided it was best to set target properties for several specific classes of alloys, as follows:

Fabricable Molybdenum
High-Strength Molybdenum

Fabricable Columbium

Unalloyed or Dilute Tungsten
High-Strength Tungsten

Tantalum

A "high strength columbium" category was initially included, but with rapid advances in the state of the art, it evolved that alloys were available which met the strength targets of the high-strength class and simultaneously satisfied the ductility targets of the fabricable class. The high-strength class was then dropped as unneeded.

The desire is to accelerate the industrial availability of alloys which have achieved a minimum developmental status, and have desirable properties; therefore, the following ground rules were established:

The following minimum billet or sheet sizes were to be produced in order to qualify for these classes:

1. Pilot Development Status

Ingots or billets: 2-inch minimum section
Sheet: 6 x 20-inch minimum size

2. Preproduction Development Status

Ingots or billets: 6-inch minimum section
Sheet: 18 x 48-inch minimum size

To insure that refractory metal alloy candidates are capable of being produced in a preproduction or production development type of contract, a demonstration of potential producibility is considered necessary. Since refractory metal alloys are, in some cases, not highly advanced, pilot development as well as preproduction development status is recognized. In order to consider the possibility of electron-beam melting columbium or tantalum alloys, 5-inch section ingots and 12 x 36-inch sheets may be taken as corresponding to preproduction status. In tungsten, sheets of 6 x 20-inch may be taken as corresponding to preproduction as well as pilot status. Sheet gages may be 0.010-inch to 0.100-inch. Although disclosure of composition is not mandatory during presentation of an alloy candidate, disclosure must be made if the alloy is selected.

In response to the desire to have a uniform basis for comparison, an evaluation group was established at the Army Materials Research Agency at Watertown. In addition, in special cases, the Defense Metals Information Center at Battelle Memorial Institute provides a uniform evaluation of weldability. In both cases, only alloys designated by the Alloy Requirements and Selection Subpanel are tested, and tests are made to confirm producers' claims before contracts are awarded.

An important accomplishment of the Alloy Requirements and Applications Subpanel is the preparation of a table of target properties. This table (Table I) provides a guide to the developers of alloys and a basis of comparison when a selection of an alloy for further development must be made. There are two classes of molybdenum alloys: fabricable and high strength. The significant difference appears in the strength and ductility requirements.

TABLE I

TABLE FOR ALLOY SELECTION

MECHANICAL PROPERTIES	Fabricable Molybdenum		High-Strength Molybdenum		Fabricable Columbium		Unalloyed or Dilute Tungsten		High-Strength Tungsten		Tantalum	
	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.
Room Temp. Tensile Ultimate Tensile Strength, ksi Yield Strength, 0.2% Offset, ksi Elong., per cent	10	10	2	2	15	15	2	2	2	2	15	15
Elevated Temp. Tensile Temp., °F Ultimate Tensile Strength, ksi Yield Strength, 0.2% Offset, ksi Elong., per cent	2000 75 60	2400 50 35	2400 75 60	2000 25 15	2000 50 40	2400 20 15	2000 20 15	2000 10 7	2000 36 24	2400 27 18	2400 35 28	2400 25 16
Creep-Rupture (State Stress and Elong.) at Temp., °F Rupture-Time, Hrs.	2000 1 10	2400 1 10	2400 1 10	3000 1 10	2000 1 10	2400 1 10	3000 1 10	3500 1 10	3000 1 10	3500 1 10	2400 3000 1 10 1 10	3500 1 10 1 10
Recrystallization (In opt. condition) 50% by met. obs. Temp., °F	1 Hr. 2400		1 Hr. 3200		1 Hr. 2400		1 Hr. 3000		1 Hr. 3400		State	State
Notch Sensitivity - ratio (a)	1.0 (RT)		1.6 (2500 F)		1.1 (RT)		1.0 (4000 F)		1.0 (400 F)		1.2 (RT)	
Transition Temp. (In Opt. Condition) in bending AT tensile, notched smooth Impact, Charpy	-40 State "		RT State "		-100 State "		+300 State "		+300 State "		-320	
Bend ductility (Room Temp.) Base metal Welded (Weld transverse to bend axis)	1 T 4 T		4 T State		1 T 2 T		4 T (300 F) State		4 T (300 F) State		1 T 2 T	
STATE FOLLOW-UP: Density Melting Point Biactivity Modulus of Elasticity Thermal Shock Resist. Creep Properties Oxidation Resistance & Corrosion Coatability Experience with 450 Brittleness Limitation Tendency												

* To be furnished

** These conditions specified by the manufacturer to yield the indicated properties.

(a) ASTM edge-notched sheet specimens, 30-40% notch depth, less than 0.001" notch radius (see ASTM Bulletin January 1960, p. 25).

The high-strength alloy is to have, at 2400 F, about the same strength as the fabricable alloy at 2000 F. As should be expected, this is paid for in part by a lower requirement for ductility. In addition to forming a basis for the selection of alloys already developed, such tables provide the producers a list of specific properties for which they should test their experimental alloys, and a specific objective for levels of properties.

The targets were submitted to the industry, and candidate alloys were screened. To date, two molybdenum alloys, both of the fabricable type, five columbium alloys, and one tantalum alloy have been selected, and sheet rolling programs on unalloyed tungsten by the powder process, by arc melting, and by shear forming are also included. Only the high-strength molybdenum and tungsten classes still await selection and contracting.

After the subpanel has determined target properties, reviewed the data presented on alloys developed by industry, and recommended those few for scaling up, it passes these conclusions to the full panel for their endorsement. At this point the military agencies take over. Provided there are no problems of policy or fund availability, bids are solicited relating to scaling up the designated alloys. It might be expected that the organization which developed the alloy would be in a preferred position, but there is normally considerable competition for the resulting contracts. The invitation to bid describes the process to be employed, the amount of metal to be processed, the size of product desired, etc. Most contracts are written with sufficient flexibility to permit the direction of work to be altered as the early results dictate.

The requirements for refractory metals change rather rapidly; therefore, the Subpanel on Alloy Requirements and Selection is continuing its review by meeting with producers of military hardware.

Consolidation and processing are the heart of the first phase of the program. A subpanel with that name has reviewed the problem broadly, looking separately at the problems of consolidation, hot working, and cold working. Specific research and development projects were outlined which could lead to improvements in quality, recovery, and cost. The discussion and conclusions are reported in MAB-179-M.

The three subpanels, Quality Specifications, Standardization of Test Methods, and Chemical Analysis, are of a slightly different character. Their functions relate more specifically to the activities of the contractors. First, when the contractor produces a sheet material in the program, it is necessary that someone tell him what quality of sheet will be accepted. This is the job of the Quality Specifications Subpanel. In a way it acts very much like the user. After weighing the difficulties of production against the requirements of the users, they have defined the minimum acceptable tensile strength, ductility, stress-rupture strength, recrystallization temperature, etc. In addition, the group specified acceptable thickness tolerance, flatness or waviness, and variations of properties within sheets and from sheet to sheet, and established a "formula" for sampling sheets to obtain the number and location of test specimens. This subpanel, in effect, states when the sheet has met the objectives of Phase I of the program, that is, when the sheet production method is satisfactory and in control, and ready

for Phases II, III and IV. The subpanel, of course, contains representation from the aeronautics and space industries who are the ultimate consumers, as well as individuals familiar with production capabilities.

The title of the subpanel, Standardization of Test Methods, is almost self descriptive. Throughout the program many tests are used to qualify a candidate alloy for the program and to determine whether the material produced in the program passes the qualifications tests. This group decides exactly how each of the tests is to be run. A survey was conducted that was intended to obtain specific recommendations from those experienced in this type of testing, and, based on this information, recommended standards have been prepared. The subpanel, of course, has reviewed and included the methods proposed by ASTM and the Titanium Sheet Rolling Program where applicable.

Report MAB-176-M describes the standardized tensile (room temperature and elevated temperature), stress rupture and creep, notched tensile, bend transition, and recrystallization tests. Work is continuing on defining other tests, particularly those which will be used in the data-collection under Phase II and Phase IV contracts, on which the properties desired by designers will be determined.

The third subpanel of this group is concerned with Chemical Analysis, and called Analytical Techniques. It is well known that analysis of the refractory metals is a difficult problem, particularly for the interstitials where, in some cases, we are now interested in quantities of less than 10 ppm. This supposition was confirmed through the results of a questionnaire, reported in MAB-178-M. This subpanel has been surveying present analytical

methods to insure that the contractors are able to provide correct analysis. They have recommended a "round robin" - at least among the contractors, and which may include many others active in refractory metals in this country, to insure reproducibility of techniques. For such a "round robin" to be meaningful, the analyses made by the various laboratories must be made on specimens which are, in fact, of the same composition. Therefore, this subpanel has recommended the preparation of "reference material" (homogeneous alloys of the appropriate nominal analysis, but whose precise composition may be uncertain) to be used for the "round robin" after suitable qualification. The ground rules for such an interlaboratory comparison were spelled out by the subpanel, and they will monitor the activity when the reference material becomes available. This subpanel also comprises members who are well aware of all similar activities in this country, such as those within ASTM.

The first objective in each of these three subpanels is to insure achievement of objectives of the Refractory Sheet Program, but certainly their results will be helpful to a broad segment of the industry as well.

Three other subpanels are again of a somewhat different nature. The program is designed to achieve high-quality, consistent, flat-rolled sheet products of certain refractory metals and their alloys. Nothing is said in this objective about coating and joining, and otherwise building the metal into a structure, but it is clear that in many applications the sheet must be coated, primarily to resist air attack, and in almost all applications it must be joined in some way. It would be tragic to produce

high-quality sheet whose use is limited because the coating, joining, and fabrication problems are unsolved. Therefore, subpanels on joining, coating and Phase III (Evaluation) were created.

The Joining Subpanel spelled out the types of fastening (with emphasis on TIG and EB welding) most promising for use with refractory metals. General recommendations are included in report MAB-171-M.

The protective coating problem is one of the most critical of all those relating to the use of refractory metal coatings. The scope of the problem was delineated through use of a comprehensive questionnaire. Results of this inquiry are reported in MAB-181-M. To permit the scaling up of promising coatings, a selection from numerous candidates must be made. Criteria for such a selection were evolved, and standardized tests (to permit inter-comparison) are being defined. The next step will be to solicit the properties of candidate coatings, make selections, and recommend that the Services award contracts for scaling up the chosen systems.

The Phase III (Fabricability Evaluation) Subpanel was set up to help inaugurate a new aspect of the over-all program. Phase I was intended to supply uniform, high quality metal. The real utility of this metal can only be demonstrated by establishing the range of conditions under which the alloys can be fabricated, and possibly by actually constructing some prototypes. The consistency of the "production" material will also be determined by making small runs of parts and measuring variations in springback, cracking, etc. The Phase III Subpanel has helped the Bureau of Aeronautics plan such a program for molybdenum and tungsten and will lay out similar programs for the other alloy bases.

In each of these subpanels, the first task has been to review the current "state of the art", and then to correlate these results with the program. A product of the subpanel activities will be recommendations for support of additional research. These research recommendations will be quite specific because they will be based upon the needs of a coordinated development activity.

The main panel coordinates all the above activities and regularly reviews contractor programs, acting as a technical advisory or consultant group for each. They are assisted in planning the agenda (especially deciding those contracts which should be reviewed) by a Steering Committee.

There are a very large number of contracts with the Services which relate in some way to refractory metal sheet. Of these, the contract officers have selected a very small number relating directly to sheet rolling to put before the panel. Some contracts have been reviewed by the panel at an early stage, but as work has proceeded which indicates that additional research is required before development can be initiated, cognizance by the panel has been dropped, at least temporarily. Although it may be impractical for the panel to monitor all the Services' contracts which are clearly devoted to refractory metal sheet rolling, it endeavors, as a minimum, to be kept informed promptly in appreciable detail. There is, of course, an obligation to avoid unintentional duplication. The task of keeping informed has been made much easier under agreements with the Defense Metals Information Center at Battelle Memorial Institute, which supplies summaries of work in progress. Two important summaries are listed in the bibliography at the end of this report.

The contracts currently of most interest to the panel are shown in Figure 2. The additional contracts which will be let will be in the areas of fabricability evaluation (proposals will soon be solicited for the evaluation of tungsten sheet), and probably also the determination of design properties of the sheet produced under the contract shown.

Figure 2

SHEET ROLLING PROJECTS

October 15, 1962

Columbium Sheet

CRUCIBLE (DuPont) (ASD)
Manufacturing Methods
X-110, D-31, F-48

FANSTEEL (BuWeps)
Pilot Production
FS-85 and B-66

WESTINGHOUSE (BuWeps)
Pilot Production
FS-85 and B-66

DU PONT (ASD)
Foil

Tantalum Sheet

WAH CHANG (ASD)
Manufacturing Methods
Ta-30Cb-7½V

WESTINGHOUSE (BuWeps)
Manufacturing Methods
8W - 2Hf

DU PONT (ASD)
Foil

Molybdenum Sheet

UNIVERSAL CYCLOPS (BuWeps)
Arc Melted Alloy
TZM and ½%Ti

MC DONNELL AIRCRAFT CO. (BuWeps)
Fabrication Evaluation
TZM and ½%Ti

Tungsten Sheet

FANSTEEL (BuWeps)
Manufacturing Methods
Undoped Sintered Tungsten

WAH CHANG
Sheet by Shear Forming
(BuWeps and ASD)

UNIVERSAL CYCLOPS (ASD)
Arc Melted Metal

DU PONT (ASD)
Foil

ADDENDUM

MATERIALS ADVISORY BOARD REPORTS RELATED TO THE
REFRACTORY METALS SHEET ROLLING PROGRAM

MAB-171-M	JOINING OF REFRACTORY SHEET METALS	March 20, 1961
MAB-172-M	REPORT ON REFRACTORY METALS SHEET ROLLING PANEL ACTIVITIES	May 22, 1961
MAB-176-M	EVALUATION TEST METHODS FOR REFRACTORY METAL SHEET MATERIALS	Sept. 6, 1961
MAB-178-M	REPORT OF THE SUBPANEL ON ANALYTICAL TECHNIQUES - REFRACTORY METALS SHEET ROLLING PANEL	Nov. 15, 1961
MAB-179-M	REPORT OF THE SUBPANEL ON CONSOLIDATION AND FABRICATION - REFRACTORY METALS SHEET ROLLING PANEL	Dec. 1, 1961
MAB-181-M	REPORT OF THE SUBPANEL ON COATINGS REFRACTORY METALS SHEET ROLLING PANEL	June 1, 1962
MAB-184-M	REPORT OF THE SUBPANEL ON QUALITY SPECIFICATIONS	June 8, 1962
MAB-164-M	(1 through 11) QUARTERLY PROGRESS REPORTS OF THE REFRACTORY METALS SHEET ROLLING PANEL (Distributed only to panel members and certain contractors and government agencies)	

OTHER PERTINENT REPORTS

DMIC Report 161	STATUS REPORT NO. 1 ON DEPARTMENT OF DEFENSE REFRACTORY METALS SHEET ROLLING PROGRAM	Nov. 2, 1961
DMIC Report 176	STATUS REPORT NO. 2 ON DEPARTMENT OF DEFENSE REFRACTORY METALS SHEET ROLLING PROGRAM	Oct. 15, 1962

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September 25, 1962

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